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**The Synthesis and Crystal Structure of LaHSe_2O_6 , a Layered,
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**The Synthesis and Crystal Structure
of LaHSe_2O_6 , a Layered, Anhydrous Selenite.**

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Abstract. Lanthanum hydrogen selenite, LaHSe_2O_6 , $M_r = 393.83$, orthorhombic, $Pc2_1b$ (No. 29), $a = 7.139(6) \text{ \AA}$, $b = 19.008(9) \text{ \AA}$, $c = 8.469(9) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 90^\circ$, $\gamma = 90^\circ$, $V = 1149.24 \text{ \AA}^3$, $Z = 8$, $D_x = 4.55 \text{ g cm}^{-3}$, $\mu = 199.7 \text{ cm}^{-1}$, $\lambda(\text{Mo K}\alpha, \text{ graphite monochromator}) = 0.71073 \text{ \AA}$, $F(000) = 1392$, room temperature (298(2) K). Final $R = 3.61\%$, $wR = 4.21\%$ for 1701 observed reflections ($I > 3\sigma(I)$). LaHSe_2O_6 has been prepared using hydrothermal synthetic techniques and its crystal structure elucidated by single crystal X-ray diffraction. This new structure consists of layers of LaO_{10} , HSeO_3 and SeO_3 polyhedra parallel to the ac -plane; the layers are interconnected by $\text{Se-OH}\cdots\text{O-}$ (Se,La) hydrogen bonds.

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Introduction. Until recently there has been very little work, structural or otherwise, reported on phases containing rare earth elements in combination with selenite (SeO_3^{2-}) anions. The structures of $\text{PrH}_3(\text{SeO}_3)_2(\text{Se}_2\text{O}_5)$ (Koskenlinna and Valkonen, 1977), CeSe_2O_6 (Delage, Carpy, H'Naifi and Gourselle, 1986) and $\text{NaLa}(\text{SeO}_3)_2$ (Morris, Hriljac and Cheetam, 1990) have been reported in the recent literature, these being the only anhydrous examples to date. These phases consist of the lanthanide atom in typical high coordination to oxygen, the coordination number depending on its ionic radius (eightfold for cerium ($r(\text{Ce}^{3+}) = 1.28 \text{ \AA}$), ninefold for praseodymium ($r(\text{Pr}^{3+}) = 1.32 \text{ \AA}$) and tenfold for lanthanum ($r(\text{La}^{3+}) = 1.40 \text{ \AA}$)), linked into a three dimensional structure *via* the sharing of faces and/or edges with other $\text{Ln}-\text{O}$ ($\text{Ln} = \text{lanthanide}$) polyhedra, and vertices with the trigonal-pyramidal SeO_3 units. This paper reports the synthesis, using hydrothermal techniques, and structure of a new layered lanthanum selenite, LaHSe_2O_6 .

Experimental. LaHSe_2O_6 was prepared from lanthanum nitrate, $\text{La}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (2 g), selenium dioxide, SeO_2 (5 g) and 15 cm^3 of water in a teflon-lined steel autoclave. The autoclave was heated to 200°C for 48 hours and then allowed to cool to room temperature. Colorless, plate-like crystals (maximum dimensions $3 \times 1 \times 0.5 \text{ mm}$) were recovered from the autoclave by suction filtration. A small crystal ($0.5 \times 0.1 \times 0.05 \text{ mm}$) was mounted on a glass fibre using cyano-acrylate glue, and intensity data were collected on an Enraf-Nonius Cad-4 diffractometer (graphite-monochromated $\text{Mo K}\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$). The unit cell constants were determined from 25 centered reflections ($12^\circ < \theta < 18^\circ$) and then refined by the method of least squares. 3750 intensity data were collected in the range $0^\circ < 2\theta < 60^\circ$, using the ω - 2θ scan mode (scan width = $(1.0 + 0.35 \tan \theta)^\circ$, minimum scan speed = $1.3^\circ \text{ min}^{-1}$, maximum scan speed = $6.7^\circ \text{ min}^{-1}$, $0 \leq h \leq 10$, $0 \leq k \leq 28$, $0 \leq l \leq 12$). The systematic absences ($0kl$, l ; $hk0$, k) were consistent with the spacegroups $Pcmb$ (No. 57; standard setting $Pbcm$) and $Pc2_1b$ (non-standard setting of $Pca2_1$, No. 29). No model could be established in the centrosymmetric spacegroup, and

all subsequent calculations assumed the structure was non-centrosymmetric, as confirmed by the successful course of the refinement. ψ -scan data on 2 reflections with $\chi \sim 90^\circ$ were recorded in order to apply an empirical absorption correction (minimum correction = 1.34, maximum correction = 7.12) and the raw data were prepared for structure solution and refinement using the program RC85 (Baird, 1987), resulting in 1701 observed reflections, according to the criterion $I > 3\sigma(I)$. After the usual corrections for Lorentz and polarization effects were made, the heavy atom positions were located from a super-sharpened Patterson function using the program SHELXS-86 (Sheldrick, 1986), and the oxygen atom positions were located by repeated Fourier difference syntheses following refinement of the known atom positions.

All the least-squares and subsidiary calculations were performed using the Oxford CRYSTALS system (Watkin, Carruthers and Betteridge, 1986) and all programs were executed on a VAX 3800 computer. The origin with respect to the polar y -direction was fixed as the centroid of the structure (Waser, 1974). The last cycle of full-matrix refinement on F (1701 reflections, 164 independent parameters, final $\sigma/\text{e.s.d.} < 0.01$), including anisotropic thermal parameters for La, Se and O, converged to final agreement factors of $R = 3.61\%$, $wR = 4.21\%$ using complex neutral-atom scattering factors taken from *International Tables for Crystallography* (1974). The weighting scheme used was that of Tukey and Prince (Carruthers and Watkin, 1979), fitted using a Chebyshev polynomial (coefficients 22(2), -17(2) and 16(2)). A final difference Fourier synthesis revealed no peaks that were refineable as the hydrogen atoms (maximum peak height $\approx 2.0 \text{ e } \text{\AA}^{-3}$, minimum $= -1.3 \text{ e } \text{\AA}^{-3}$).

Discussion. Table 1* gives the final atomic positional and thermal parameters and Table 2 gives selected bond distances and angles for the structure. The structure of LaHSe_2O_6 (Figures 1 and 2) consists of layers of LaO_{10} and $(\text{H})\text{SeO}_3$ groups parallel to the ac plane, these sheets being held together through hydrogen bonding. The structure contains two

crystallographically-independent lanthanum atoms, both of which are deca-coordinated to oxygen in irregular geometry (ave. La(1)-O distance = 2.615(3) Å, ave. La(2)-O distance = 2.622(3) Å), similar to that found in NaLa(SeO₃)₂ ($d_{\text{ave}}(\text{La-O}) = 2.635(3) \text{ Å}$) (Morris, Hriljac and Cheetham, 1990), and four crystallographically-distinct selenium atoms, all of which form their characteristic pyramidal coordination with three oxygen atom neighbors. The twelve crystallographically-distinct oxygen atoms can be classified into four groups with respect to the number and types of their nearest neighbour atoms: Six oxygen atoms; O(1), O(2), O(4), O(6), O(8) and O(10), which are coordinated with two lanthanum and one selenium atom each, atoms O(3) and O(5), are coordinated with three lanthanum atoms and one selenium atom; atoms O(7) and O(11) bond to one lanthanum atom and one selenium atom. Two more atoms, O(9) and O(12), which are protonated (confirmed by bond-strength/bond-length analysis) are coordinated with a selenium atom. The possible H-bonding scheme is discussed below.

The lanthanum atoms are both shifted from the centroids of their coordinating-oxygen polyhedra; La(1) by 0.15(1) Å and La(2) by 0.11(1) Å, the distortion being largely in the direction of the polar axis of the unit cell (i.e. the b-axis). The program MISSYM (Le Page, 1988) which checks a crystallographic atomic model for 'missing' (unmodelled) symmetry, did not detect that this polyhedral asymmetry was the result of a distortion from a possible centrosymmetric prototype structure, allowing all the atoms to move up to 1 Å to assume their possible new positions. All four selenium atoms exhibit the usual trigonal pyramidal geometry with an (unobserved) stereochemically active lone pair of electrons (ave. Se-O distance = 1.702(3) Å, O-Se-O bond angle average = 99.3(2)°). These values compare satisfactorily with the average geometrical parameters ($\langle d(\text{Se-O}) \rangle = 1.709(10) \text{ Å}$, $\langle \theta(\text{O-Se-O}) \rangle = 100.2(1.3)^\circ$; values in parentheses = r.m.s. deviations) derived by Hawthorne, Groat and Ercit (1987) for a large number of (H)SeO₃ species.

One $\text{La}(1)\text{O}_{10}$ polyhedron is linked via the sharing of two triangular faces with two other $\text{La}(2)\text{O}_{10}$ units, and with two more $\text{La}(1)\text{O}_{10}$ groups via the sharing of edges, forming an infinite layer. The layers incorporate the selenium atoms in two ways: $\text{Se}(1)$ and $\text{Se}(2)$ share all three oxygen atom vertices with neighbouring LaO_{10} units, while $\text{Se}(3)$ and $\text{Se}(4)$ share only two of their oxygen vertices with lanthanum atoms. The two remaining oxygens of the $\text{Se}(3)$ and $\text{Se}(4)$ selenite groups ($\text{O}(8)$ and $\text{O}(10)$ respectively) are protonated and point into the interlamellar region, and it is through these atoms that the hydrogen bonding which binds the layers together occurs. The proton positions were not located in the diffraction study, but reasonable inter-layer H-bonding oxygen-oxygen atom distances (indicated by ...) include $\text{O}(9)\text{H}\cdots\text{O}(11)$ ($d(\text{O}-\text{O}) = 2.541(15) \text{ \AA}$) and $\text{O}(12)\text{H}\cdots\text{O}(9)$ ($d(\text{O}-\text{O}) = 2.686(15) \text{ \AA}$). Assuming an $\text{O}-\text{H}$ distance of 1.0 \AA , and linear $\text{O}-\text{H}-\text{O}$ bonds between layers, $\text{Se}-\text{O}-\text{H}$ angles of 106 and 118° would result from these interactions. Both these H-bonds also lead to short non-bonding Se to H distances of less than 2.4 \AA .

This compound is the first layered anhydrous selenite to be discovered, complementing the layered praeodymium selenite/diselenite phase $\text{PrH}_3(\text{SeO}_3)_2(\text{Se}_2\text{O}_5)$ (Koskenlinna and Valkonen, 1977), and layered hydrated selenite phases such as $\text{CaSeO}_3 \cdot \text{H}_2\text{O}$ (Hawthorne, Ercit and Groat, 1986) and the recently discovered $\text{LnHSe}_2\text{O}_6 \cdot 2\text{H}_2\text{O}$, $\text{Ln} = \text{Y}, \text{Pr}-\text{Yb}$ (except Pm) (Morris *et al.* 1991). The 2-dimensional structure of LaHSe_2O_6 , which is layered with respect to the polar crystallographic direction, combined with the acidic protons which must be present, leads to interesting possibilities for ion exchange and intercalation chemistry. Studies of these reactions are now being undertaken.

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* Lists of observed and calculated structure factors and anisotropic thermal parameters

have been deposited with the British Library Document Supply Center as Supplementary Publication No. (pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester, CH1 2HU, England.

Figure Captions:

Figure 1: Detail of the La/Se/O sheet in LaHSe_2O_6 showing the atom labelling scheme. Thermal ellipsoids are at the 50% probability level.

Figure 2: Packing diagram for LaHSe_2O_6 , viewed down the *a*-direction, showing the proposed inter-layer H-bonding scheme, indicated by thin lines.

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Table 1: Atomic Positional Parameters



Atom	x	y	z	U_{equiv}^\dagger
La(1)	0.41902(8)	0.4108(1)	0.84339(7)	0.0082
La(2)	0.91735(8)	0.4997(1)	0.94737(7)	0.0082
Se(1)	0.9347(1)	0.3586(2)	0.6924(1)	0.0084
Se(2)	0.4304(1)	0.5516(2)	1.0944(1)	0.0089
Se(3)	0.4390(2)	0.2601(2)	0.6264(2)	0.0140
Se(4)	0.9217(2)	0.6508(2)	1.1708(1)	0.0144
O(1)	0.768(1)	0.3923(5)	0.817(1)	0.0064
O(2)	0.863(1)	0.3826(6)	0.509(1)	0.0151
O(3)	1.097(1)	0.4293(4)	0.715(1)	0.0063
O(4)	0.361(1)	0.5316(6)	1.277(1)	0.0161
O(5)	0.593(1)	0.4873(5)	1.075(1)	0.0096
O(6)	0.264(1)	0.5217(5)	0.971(1)	0.0195
O(7)	0.367(2)	0.2751(5)	0.811(1)	0.0124
O(8)	0.472(1)	0.3473(5)	0.581(1)	0.0110
O(9)	0.672(2)	0.2366(6)	0.665(1)	0.0156
O(10)	0.983(1)	0.5719(5)	1.210(1)	0.0071
O(11)	0.816(2)	0.6383(6)	0.998(1)	0.0216
O(12)	1.129(2)	0.6878(7)	1.089(1)	0.0300

$$^\dagger U_{\text{equiv}} = (U_1 U_2 U_3)^{1/3}$$

Table 2: Bond Distances(Å)/Angles(°)**LaHSe₂O₆**

La(1)—O(1)	2.528(8)	La(1)—O(2 ^a)	2.51(1)
La(1)—O(3 ^b)	2.571(7)	La(1)—O(4 ^c)	2.84(1)
La(1)—O(5)	2.738(9)	La(1)—O(5 ^c)	2.702(9)
La(1)—O(6)	2.61(1)	La(1)—O(7)	2.622(9)
La(1)—O(8)	2.55(1)	La(1)—O(8 ^a)	2.477(9)
La(2)—O(1)	2.553(9)	La(2)—O(2 ^d)	2.77(1)
La(2)—O(3)	2.702(8)	La(2)—O(3 ^d)	2.632(8)
La(2)—O(4 ^c)	2.53(1)	La(2)—O(5)	2.564(8)
La(2)—O(6 ^e)	2.52(1)	La(2)—O(10)	2.658(9)
La(2)—O(10 ^f)	2.533(9)	La(2)—O(11)	2.76(1)
Se(1)—O(1)	1.713(8)	Se(1)—O(2)	1.697(9)
Se(1)—O(3)	1.783(8)		
Se(2)—O(4)	1.671(9)	Se(2)—O(5)	1.694(8)
Se(2)—O(6)	1.68(1)		
Se(3)—O(7)	1.67(1)	Se(3)—O(8)	1.717(9)
Se(3)—O(9)	1.76(1)		
Se(4)—O(10)	1.599(9)	Se(4)—O(11)	1.66(1)
Se(4)—O(12)	1.78(1)		

O(2 ^a)-La(1)-O(1)	144.1 (3)
O(3 ^b)-La(1)-O(2 ^a)	63.2 (3)
O(4 ^c)-La(1)-O(2 ^a)	136.6 (4)
O(5)-La(1)-O(1)	71.9 (3)
O(5)-La(1)-O(3 ^b)	129.6 (3)
O(5 ^c)-La(1)-O(1)	91.9 (3)
O(5 ^c)-La(1)-O(3 ^b)	62.7 (2)
O(5 ^c)-La(1)-O(5)	109.4 (3)
O(6)-La(1)-O(2 ^a)	66.6 (3)
O(6)-La(1)-O(4 ^c)	70.4 (3)
O(7)-La(1)-O(1)	89.7 (3)
O(7)-La(1)-O(3 ^b)	87.9 (3)
O(7)-La(1)-O(5)	131.5 (3)
O(8)-La(1)-O(2 ^a)	120.2 (3)
O(8)-La(1)-O(4 ^c)	97.4 (3)
O(8 ^a)-La(1)-O(1)	72.1 (3)
O(8 ^a)-La(1)-O(3 ^b)	133.9 (3)
O(8 ^a)-La(1)-O(5)	62.2 (3)
O(7)-La(1)-O(5 ^c)	115.9 (3)
O(8)-La(1)-O(5 ^c)	61.8 (3)
O(8)-La(1)-O(7)	57.6 (3)
O(8 ^a)-La(1)-O(6)	101.0 (3)
O(8 ^a)-La(1)-O(8)	115.5 (3)

O(2 ^d)-La(2)-O(1)	71.1 (3)
O(3)-La(2)-O(2 ^d)	58.2 (3)
O(3 ^d)-La(2)-O(2 ^d)	56.7 (2)
O(4 ^c)-La(2)-O(1)	67.5 (3)
O(4 ^c)-La(2)-O(3)	94.3 (3)
O(5)-La(2)-O(1)	74.5 (3)
O(5)-La(2)-O(3)	133.5 (3)
O(5)-La(2)-O(4 ^c)	63.4 (3)
O(6 ^e)-La(2)-O(2 ^d)	64.0 (3)
O(6 ^e)-La(2)-O(3 ^d)	93.0 (3)

O(3 ^b)-La(1)-O(1)	149.9 (3)
O(4 ^c)-La(1)-O(1)	63.2 (3)
O(4 ^c)-La(1)-O(3 ^b)	107.6 (3)
O(5)-La(1)-O(2 ^a)	94.4 (3)
O(5)-La(1)-O(4 ^c)	57.3 (3)
O(5 ^c)-La(1)-O(2 ^a)	124.1 (3)
O(5 ^c)-La(1)-O(4 ^c)	54.3 (2)
O(6)-La(1)-O(1)	124.5 (3)
O(6)-La(1)-O(3 ^b)	71.7 (3)
O(6)-La(1)-O(5)	57.9 (3)
O(7)-La(1)-O(2 ^a)	74.6 (3)
O(7)-La(1)-O(4 ^c)	148.7 (3)
O(8)-La(1)-O(1)	73.3 (3)
O(8)-La(1)-O(3 ^b)	80.1 (3)
O(8)-La(1)-O(5)	143.7 (3)
O(8 ^a)-La(1)-O(2 ^a)	72.1 (3)
O(8 ^a)-La(1)-O(4 ^c)	112.3 (3)
O(6)-La(1)-O(5 ^c)	84.3 (3)
O(7)-La(1)-O(6)	140.9 (3)
O(8)-La(1)-O(6)	143.4 (3)
O(8 ^a)-La(1)-O(5 ^c)	163.4 (3)
O(8 ^a)-La(1)-O(7)	69.5 (3)

O(3)-La(2)-O(1)	59.1 (3)
O(3 ^d)-La(2)-O(1)	87.2 (3)
O(3 ^d)-La(2)-O(3)	113.2 (3)
O(4 ^c)-La(2)-O(2 ^d)	138.2 (4)
O(4 ^c)-La(2)-O(3 ^d)	125.7 (3)
O(5)-La(2)-O(2 ^d)	111.0 (3)
O(5)-La(2)-O(3 ^d)	63.7 (2)
O(6 ^e)-La(2)-O(1)	125.2 (3)
O(6 ^e)-La(2)-O(3)	71.0 (3)
O(6 ^e)-La(2)-O(4 ^c)	141.0 (3)

O(10)–La(2)–O(1)	148.1(3)	O(10)–La(2)–O(2 ^d)	98.9(3)
O(10)–La(2)–O(3)	141.4(3)	O(10)–La(2)–O(3 ^d)	63.1(3)
O(10)–La(2)–O(4 ^c)	119.6(3)	O(10 ^f)–La(2)–O(1)	101.9(3)
O(10 ^f)–La(2)–O(2 ^d)	115.2(3)	O(10 ^f)–La(2)–O(3 ^e)	63.8(3)
O(10 ^f)–La(2)–O(3 ^d)	165.4(3)	O(10 ^f)–La(2)–O(4 ^c)	68.7(3)
O(11)–La(2)–O(1)	136.1(3)	O(11)–La(2)–O(2 ^d)	152.0(3)
O(11)–La(2)–O(3)	135.0(3)	O(11)–La(2)–O(3 ^d)	110.0(3)
O(11)–La(2)–O(4 ^c)	69.8(3)	O(6 ^e)–La(2)–O(5)	150.3(3)
O(10)–La(2)–O(5)	81.7(3)	O(10)–La(2)–O(6 ^e)	70.9(3)
O(10 ^f)–La(2)–O(5)	129.4(3)	O(10 ^f)–La(2)–O(6 ^e)	72.4(3)
O(10 ^f)–La(2)–O(10)	109.6(3)	O(11)–La(2)–O(5)	77.7(3)
O(11)–La(2)–O(6 ^e)	94.9(3)	O(11)–La(2)–O(10)	54.9(3)
O(11)–La(2)–O(10)	71.3(3)		
O(2)–Se(1)–O(1)	104.6(5)	O(3)–Se(1)–O(1)	95.9(4)
O(3)–Se(1)–O(2)	95.2(4)		
O(5)–Se(2)–O(4)	97.6(5)	O(6)–Se(2)–O(4)	106.8(5)
O(6)–Se(2)–O(5)	100.3(4)		
O(8)–Se(3)–O(7)	94.9(5)	O(9)–Se(3)–O(7)	99.3(6)
O(9)–Se(3)–O(8)	99.1(5)		
O(11)–Se(4)–O(10)	100.1(5)	O(12)–Se(4)–O(10)	103.0(6)
O(12)–Se(4)–O(11)	95.1(6)		

Symmetry codes: (a) $1-x, y, \frac{1}{2}+z$; (b) $-1+x, y, z$; (c) $1-x, y, -\frac{1}{2}+z$; (d) $2-x, y, \frac{1}{2}+z$; (e) $1+x, y, z$; (f) $2-x, y, -\frac{1}{2}+z$.

**The Synthesis and Crystal Structure
of LaHSe_2O_6 , a Layered, Anhydrous Selenite.**

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*Supplementary Material
Anisotropic thermal factors,
observed and calculated structure factor moduli.*

The temperature factor is given by: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* \cos \gamma^* + 2U_{13}hla^*c^* \cos \beta^* + 2U_{23}klb^*c^* \cos \alpha^*)]$, where the U_{ij} are mean-square amplitudes of vibration in angstroms.

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